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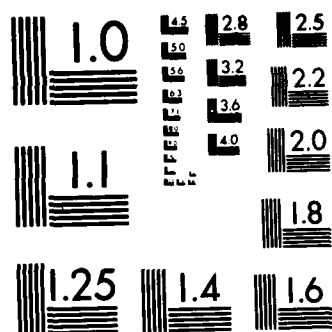
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TECHNICAL REPORT # 58

Surface Infrared Spectroelectrochemistry. The Interaction
of the Electric Field in the Electrical Double Layer with
Pyrene Adsorbed on a Platinum Electrode: Effects on the
Infrared Surface Difference Spectrum

By

Stanley Pons
C. Korzeniewski

Prepared for Publication in
Langmuir

University of Utah
Department of Chemistry
Salt Lake City, Utah 84112

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Surface Infrared Spectroelectrochemistry. The Interaction of the Electric Field in the Electrical Double Layer with Pyrene Adsorbed on a Platinum Electrode: Effects on the Infrared Surface Difference Spectrum

Carol Korzeniewski and Stanley Pons*

Department of Chemistry, University of Utah, Salt Lake City, Utah 84112

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The surface infrared spectrum of pyrene adsorbed at a platinum electrode is obtained by using the SNIFTIRS technique. Activation of Raman modes by coupling of the polarizable electrons in the molecule to the strong electric field which exists across the electrical double layer is reported. SNIFTIRS spectra are compared to both the solution IR and Raman spectra.

Introduction

The perturbation of infrared spectra by strong electric fields was suggested by Condon in 1932.¹ It was predicted that a strong static external electric field could distort polarizable electrons in a molecule in the same way that the electric vector of a light wave induced a dipole moment in symmetric molecules, effects that could be observed by Raman spectroscopy. The selection rules for absorption of infrared radiation require that the vibrational mode give rise to a dipole moment oscillating at the frequency of the exciting radiation. Application of a static electric field can distort polarizable electrons in a molecule resulting in an induced dipole moment. Oscillation of this dipole in response to motion of the nuclei symmetrically about the molecular center of mass can be detected by using infrared radiation. Thus, for highly polarizable molecules, in a strong static electric field, it is possible to detect totally symmetric (A_g) vibrational modes by infrared spectroscopy. Field-induced infrared absorption of this type has been shown convincingly for molecular hydrogen² and crystals of diamond type.³

Other types of perturbations caused by static electric fields have been observed in vibrational spectra. Changes in the intensity of infrared-active bands have been observed both in gas phase as well as electrochemical systems.⁴ The potential dependence of vibrational frequency has also been investigated.⁵⁻⁷ Attempts have been made to give a more quantitative explanation of the role of the electric field in such systems.⁸⁻¹²

Since electric fields on the order of 10^7 V/m exist across the polarized electrode/solution interface,¹³ an electrochemical system with absorbers in the interfacial region is ideally suited to study the effects of strong electric fields on infrared spectra. Recently, several techniques have been developed to obtain spectra of species in the electrical double layer.^{14,15} In this report the SNIFTIRS (subtractively normalized interfacial Fourier transform infrared spectroscopy) technique has been used to observe infrared absorption of pyrene at frequencies where, under external field free conditions, only Raman modes are active.

Experimental Section

Electrochemical cells used in the experiments were constructed from glass syringes (JAS Instrument Systems, Inc.). An infrared-transparent window (50 mm diameter \times 0.3 mm thickness) made of single-crystal n-type silicon was fitted on the front of the cell. The working electrode was constructed from a platinum disk (9 mm diameter \times 1 mm thick) sealed in glass and polished to a mirror finish with alumina of decreasing sizes down to 0.05 μ m. Final polishing was made from a balsa wood base to minimize the embedding of alumina particles in the platinum surface. Cells were cleaned by acid treatment (50/50 vol % sulfuric/nitric acid) followed by rinsing in triply distilled water.

Acetonitrile (Burdick and Jackson, Muskegon, MI) containing less than 0.009% (nominal) water was handled under a dry argon atmosphere. Pyrene was obtained from Aldrich and purified by vacuum sublimation. Tetra-n-butylammonium tetrafluoroborate

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4 (TBAF) was prepared by the metathesis of sodium tetrafluoro-
12 borate (Aldrich) and tetra-*n*-butylammonium hydrogen sulfate
18 (Aldrich). It was dried in vacuo at 80 °C for a minimum of 24
15 h before use.

PAR18

SEN03 1 The electrochemistry was controlled by a potentiostat and
SEN06 10 waveform generator (Hi-Tek Instruments). The instrumentation
4 for phase-sensitive detection used in the differential capacity
SEN08 12 measurements was obtained from Bentham Instruments. Infrared
3 spectra were obtained with the use of an IBM IR/98 Series FTIR
SEN12 15 spectrometer. Light from the source was focused onto the electro-
SEN15 10 de and then reflected out of the cell onto the detector. The
2 spectrum was obtained by collecting interferograms at two
SEN18 11 electrode potentials, E_1 and E_2 . After transformation of the
6 interferograms to the frequency domain, the two spectra were
16 ratioed to give R_2/R_1 , where the R_i correspond to the reflectances
SEN21 26 at each potential E_i . The spectra are normally displayed as R_2/R_1
9 on the spectrometer, and may be converted to the more common
SEN24 20 units of $\Delta R/R$ through the relation $\Delta R/R = R_2/R_1 - 1$. This final
4 result represents the difference spectrum of the species being
13 observed between the two potentials.

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SEN03 1 **Results**
13 Figure 1 is a plot of the differential capacity of the
21 platinum electrode used in the SNIFTIRS experiments in
SEN06 24 acetonitrile solutions (a) without and (b) with pyrene.
11 Addition of pyrene to the system significantly lowers the
17 differential capacitance of the platinum electrode indi-
SEN09 18 cating adsorption of pyrene across the entire potential
7 range investigated. Reduction at large negative potentials
SEN12 18 results in the desorption of the species and increase in the
8 capacitance as expected. Since we are working at pyrene
16 concentrations below 0.5 mM, it is likely that the pyrene
is adsorbed in a flat orientation.

PAR24

SEN03 1 Figure 2 shows the electrochemical behavior of pyrene
SEN06 10 in acetonitrile. It is noted that the current is constant and
11 essentially capacitive over the region of +0.5 to -1.5 V (vs.
SEN09 22 Ag/Ag⁺). Reduction of pyrene occurs at potentials more
SEN12 9 negative than -2.4 V. To study the effect that the electric
10 field has on the spectra independent of chemical effects
18 such as reduction of pyrene, we have collected interfero-
SEN15 26 grams at potentials between +0.5 and -1.5 V. Thus, any
15 changes in the spectra are due to changes induced by the
electric field rather than a chemical change.

PAR27

SEN03 1 Figure 3 shows SNIFTIRS spectra in the region of the
12 ring stretching modes of pyrene as a function of the in-
SEN06 22 tensity of potential modulation. A band, 1640 cm⁻¹ is ob-
7 served, which is weakly dependent on the magnitude of
SEN09 18 the electrode potential. The position of the band appears
8 to shift to higher energy as the electrode potential is made
SEN12 19 more positive. This result was reproducible in several
SEN15 8 independent experiments. The SNIFTIRS spectra can be
7 compared to the Raman (Figure 4) and infrared (Figure
SEN18 16 5) solution spectra. The bands in the Raman spectrum
8 are assigned to symmetric C-C stretching ring modes
SEN21 16 (A_g).¹⁶ The infrared spectrum has a strong band at 1598
11 cm⁻¹ assigned to the B_{3g} ring mode and a strong band at
23 3049 cm⁻¹ assigned to the aromatic C-H stretch.¹⁶

PAR30

SEN03 1 Figure 6 is the SNIFTIRS difference spectra of the same
12 pyrene platinum electrode system in the region of the
SEN06 21 pyrene aromatic C-H stretch. We note the absence of any
8 SNIFTIRS absorption bands in the 3049-cm⁻¹ region.

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SEN03 1 **Discussion**
9 Pyrene was chosen to study field-induced infrared ab-
17 sorption because of its large molecular polarizability¹⁷ and
25 the likelihood that the molecule would undergo flat ad-
SEN06 34 sorption on the platinum electrode under low bulk solution
SEN09 6 conditions (vide infra). Measurements have been made
11 to ensure adsorption and flat orientation of pyrene. The
differential capacity of the electrode indicates that pyrene
SEN12 11 is adsorbed over the potential range studied. Further, we
4 are working at concentrations where pyrene should adsorb
SEN15 12 flat on the surface rather than edgewise. Soriaga and
4 Hubbard have studied the concentration dependence of

FIG 1 (003, 3- 4)

FIG 2 (003, 3- 4)

FIG 3 (003, 3- 4)

FIG 4 (015,12-13)
FIG 5 (015,16-17)

FNT 16

FIG 6 (003, 3- 4)

FNT 17-20

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11 the orientation of several π -electron-rich molecules ad-
SEN18 17 sorbed at platinum electrodes.²¹ They have shown that
6 in general, flat adsorption is the primary surface configura-
14 tion at bulk concentrations less than about 0.5 mM.

PAR34

SEN03 1 The absence of an absorption band in the SNIFTIRS
11 spectra of Figure 6 provides further evidence that pyrene
SEN06 20 is orientated flat on the surface. If the molecule were
6 orientated edgewise on the electrode a strong band at
SEN09 23 about 3049 cm^{-1} corresponding to the aromatic C-H
8 stretch would be expected to appear. However, if the
SEN12 13 molecule were orientated flat on the surface absorption
SEN15 7 would be forbidden. This differential absorption is known
6 as the surface selection rule. The surface selection rule
SEN18 16 arises due to the properties of radiation reflection from a
8 metal surface. These properties may be derived through
SEN21 17 analysis of the Fresnel relations and have been discussed
7 in detail elsewhere.¹⁸ When infrared radiation is reflected
16 from a metal surface, only the component polarized parallel
SEN24 26 to the plane of incidence (p polarized) has any amplitude
4 at the surface after reflection. Light polarized perpen-
13 dicular to the plane of incidence (s polarized) undergoes
26 a phase shift of close to 180° for all angles of incidence
SEN27 35 resulting in a standing wave that has little amplitude near
8 the surface. Infrared radiation will interact with an os-
19 cillating dipole of a species when both the electric field of
SEN30 27 the radiation and the oscillator have spatial components
6 in the same direction. Thus, only molecules that have a
17 component of the dipole derivative (the change in the
26 dipole moment with respect to the normal coordinate)
SEN33 34 oriented in a direction perpendicular to the surface can
SEN36 4 interact with the p-polarized radiation. The s-polarized
12 radiation is blind to species adsorbed near the surface. For
21 a molecule adsorbed flat on the surface, absorption of
30 infrared radiation is forbidden by the surface selection rule.
SEN39 1 However, if a dipole moment is induced in the species
21 perpendicular to the surface, for example, by external fields
or bonding effects, a vibrational transition can be observed
by using infrared radiation.

PAR39

SEN03 1 The appearance of symmetry-forbidden bands in the
9 spectra of molecules adsorbed on metal surfaces has been
SEN06 18 observed. The interpretation of such bands includes
8 mechanisms involving chemical bonding of the molecule
15 to the surface and interaction of the molecule with electric
SEN09 25 fields near the metal surface. The chemical mechanism
6 suggests that bonding to the surface decreases the sym-
13 metry of the molecule causing disallowed modes to become
SEN12 22 active.¹⁹ In addition, distortion of the molecule by dona-
9 tion of electrons from the metal to orbitals on the molecule
SEN15 20 has also been suggested. More quantitative explanations
5 based on electric fields present near the metal surface have
SEN18 16 been discussed. Sass et al.²⁰ have shown that electric field
18 gradients arising from interaction of radiation with the
metal surface are strong enough to couple with quadrupole
SEN21 26 moments in the molecule, giving rise to activation of in-
7 frared-forbidden modes. In electrochemical systems, it has
15 been shown quantitatively that large electric fields which
24 exist across the electrical double layer are strong enough
SEN24 1 to interact with electrons of highly polarizable molecules.
SEN27 11 This interaction results in a dipole moment which can
4 oscillate normal to the metal surface.⁹ Applying this
SEN30 15 calculation⁹ to pyrene predicts a $\Delta R/R$ on the order of 10^{-4}
14 for an electric field strength of 10^6 V/cm. Therefore, we
22 believe that the mechanism for appearance of bands in the
SEN33 30 SNIFTIRS difference spectrum of Figure 3 is through
3 interaction of polarizable electrons in the molecule with
12 the large static electric field across the double layer. This
SEN36 11 interaction can induce a dipole moment normal to the
8 surface which can oscillate at the vibrational frequency of
18 the A_g ring mode. The band appears at potentials very
close in energy to those observed for ring stretching modes
in the Raman spectrum (which are infrared-forbidden).

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SEN03 1 Thus, for adsorbed pyrene a dipole moment can be in-
11 duced normal to the surface by coupling the highly po-
20 larizable electrons in the aromatic ring of the molecule to

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SEN06 30 the electric field across the double layer. The aromatic
4 C-H stretching modes would not be expected to be en-
13 hanced by the electric field because of the small polariz-
22 ability of the C-H bond.

TXT15

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SEN03 1

Conclusion

11 This report demonstrates that the electric field in the
12 double layer is sufficiently strong to induce infrared ac-
13 tivity in modes which are forbidden by normal infrared
14 selection rules. Field induced absorption can be used to
15 study the electric field in the double layer.

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many helpful discussions of the work, and we also ac-
knowledge the assistance of and discussion with Dr. Mi-
chael Hunnicutt and Professor Joel M. Harris regarding
pyrene. These workers have investigated similar effects
of pyrene adsorbed at dielectric surfaces.²²

*To whom correspondence should be addressed.

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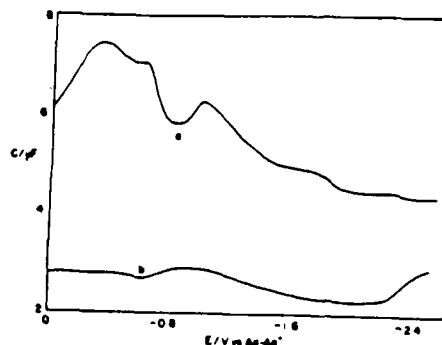


Figure 1. Differential capacity curves for a platinum electrode in a solution of (a) 0.1 M TBAF in acetonitrile and (b) same as (a) with 0.5 mM pyrene. Results were obtained by using a 5 mV (pp amplitude) 400 Hz sine wave superimposed on a 10 mV/s voltage ramp applied to the working electrode and measuring the in and out of phase components of the ac current. All potentials are with reference to the Ag/Ag⁺ (0.01 M Ag⁺ in acetonitrile with 0.1 M TBAF).

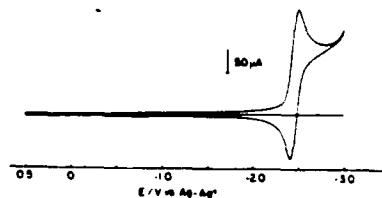


Figure 2. Cyclic voltammogram of the solution in Figure 1b. Sweep rate is 50 mV/s.

except with 5 mM PYRENE

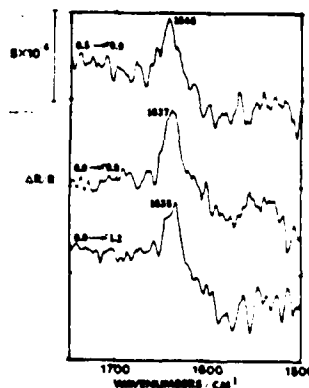
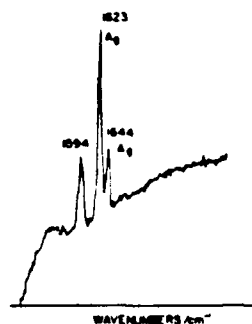
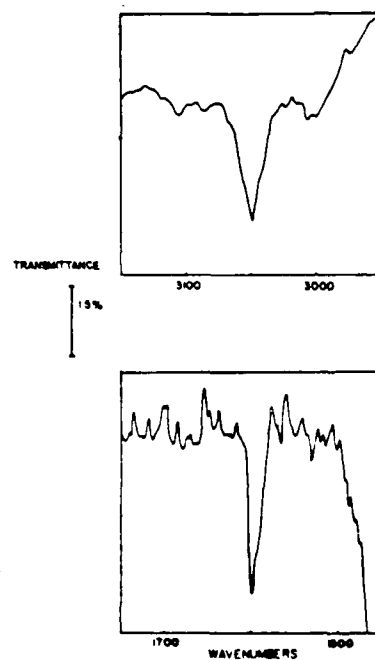


Figure 3. SNIPTIRS difference spectrum of the solution in Figure 1b between the indicated potential regions. Bands extending down are present increased absorbance at the more negative electrode potential.

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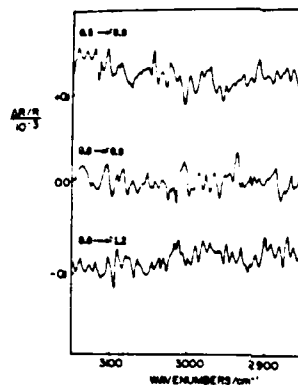


CAPO0 1 Figure 4. Solution Raman spectrum of pyrene in carbon tet-
 CAPO6 9 rachloride. Band assignments are taken from ref 16



CAPO0 1 Figure 5. Solution infrared spectrum of pyrene in acetonitrile.
 CAPO6 10 (a) The ring stretching mode region. (b) The aromatic C-H stretch
 CAPO9 7 region (see ref 16).

FIG. 3
FN. 56



CAP00 1 **Figure 6.** SNIPTIRS difference spectrum in the aromatic C-H
CAP03 9 stretch region for the solution described in Figure 1b.

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Korzeniewski and Pons

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Dr. Robert Somoano
Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California 91103

Dr. Johann A. Joebstl
USA Mobility Equipment R&D Command
DRDME-EC
Fort Belvoir, Virginia 22060

Dr. Judith H. Ambrus
NASA Headquarters
M.S. RTS-6
Washington, D.C. 20546

Dr. Albert R. Landgrebe
U.S. Department of Energy
M.S. 68025 Forrestal Building
Washington, D.C. 20595

Dr. J. J. Brophy
Department of Physics
University of Utah
Salt Lake City, Utah 84112

Dr. Charles Martin
Department of Chemistry
Texas A&M University
College Station, Texas 77843

Dr. H. Tachikawa
Department of Chemistry
Jackson State University
Jackson, Mississippi 39217

Dr. Theodore Beck
Electrochemical Technology Corp.
3935 Leary Way N.W.
Seattle, Washington 98107

Dr. Farrell Lytle
Boeing Engineering and
Construction Engineers
P.O. Box 3707
Seattle, Washington 98124

Dr. Robert Gotscholl
U.S. Department of Energy
MS G-226
Washington, D.C. 20545

Dr. Edward Fletcher
Department of Mechanical Engineering
University of Minnesota
Minneapolis, Minnesota 55455

Dr. John Fontanella
Department of Physics
U.S. Naval Academy
Annapolis, Maryland 21402

Dr. Martha Greenblatt
Department of Chemistry
Rutgers University
New Brunswick, New Jersey 08903

Dr. John Wasson
Syntheco, Inc.
Rte 6 - Industrial Pike Road
Gastonia, North Carolina 28052

Dr. Walter Roth
Department of Physics
State University of New York
Albany, New York 12222

Dr. Anthony Sammells
Eltron Research Inc.
4260 Westbrook Drive, Suite 111
Aurora, Illinois 60505

Dr. C. A. Angell
Department of Chemistry
Purdue University
West Lafayette, Indiana 47907

Dr. Thomas Davis
Polymer Science and Standards
Division
National Bureau of Standards
Washington, D.C. 20234

Ms. Wendy Parkhurst
Naval Surface Weapons Center R-33
R-33
Silver Spring, Maryland 20910

DL/413/83/01
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ABSTRACTS DISTRIBUTION LIST, 359/627

Dr. John Owen
Department of Chemistry and
Applied Chemistry
University of Salford
Salford M5 4WT ENGLAND

Dr. Boone Owens
Department of Chemical Engineering
and Materials Science
University of Minnesota
Minneapolis, Minnesota 55455

Dr. J. O. Thomas
University of Uppsala
Institute of Chemistry
Box 531
S-751 21 Uppsala, Sweden

Dr. O. Stafsudd
Department of Electrical Engineering
University of California
Los Angeles, California 90024

Dr. S. G. Greenbaum
Department of Physics
Hunter College of CUNY
New York, New York 10021

Dr. Menahem Anderman
W.R. Grace & Co.
Columbia, Maryland 20144

ABSTRACTS DISTRIBUTION LIST, 359/627

Dr. Hector D. Abruna
Department of Chemistry
Cornell University
Ithaca, New York 14853

Dr. A. B. P. Lever
Chemistry Department
York University
Downsview, Ontario M3J1P3

Dr. Stanislaw Szpak
Naval Ocean Systems Center
Code 633, Bayside
San Diego, California 95152

Dr. Gregory Farrington
Department of Materials Science
and Engineering
University of Pennsylvania
Philadelphia, Pennsylvania 19104

M. L. Robertson
Manager, Electrochemical
and Power Sources Division
Naval Weapons Support Center
Crane, Indiana 47522

Dr. T. Marks
Department of Chemistry
Northwestern University
Evanston, Illinois 60201

Dr. Micha Tomkiewicz
Department of Physics
Brooklyn College
Brooklyn, New York 11210

Dr. Lesser Blum
Department of Physics
University of Puerto Rico
Rio Piedras, Puerto Rico 00931

Dr. Joseph Gordon, II
IBM Corporation
5600 Cottle Road
San Jose, California 95193

Dr. Nathan Lewis
Department of Chemistry
Stanford University
Stanford, California 94305

Dr. D. H. Whitmore
Department of Materials Science
Northwestern University
Evanston, Illinois 60201

Dr. Alan Bewick
Department of Chemistry
The University of Southampton
Southampton, SO9 5NH ENGLAND

Dr. E. Anderson
NAVSEA-56Z33 NC #4
2541 Jefferson Davis Highway
Arlington, Virginia 20362

Dr. Bruce Dunn
Department of Engineering &
Applied Science
University of California
Los Angeles, California 90024

Dr. Elton Cairns
Energy & Environment Division
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

Dr. Richard Pollard
Department of Chemical Engineering
University of Houston
Houston, Texas 77004

Dr. M. Philpott
IBM Corporation
5600 Cottle Road
San Jose, California 95193

Dr. Donald Sandstrom
Boeing Aerospace Co.
P.O. Box 3999
Seattle, Washington 98124

Dr. Carl Kannewurf
Department of Electrical Engineering
and Computer Science
Northwestern University
Evanston, Illinois 60201

Dr. Joel Harris
Department of Chemistry
University of Utah
Salt Lake City, Utah 84112

ABSTRACTS DISTRIBUTION LIST, 359/627

Dr. M. Wrighton
Chemistry Department
Massachusetts Institute
of Technology
Cambridge, Massachusetts 02139

Dr. B. Stanley Pons
Department of Chemistry
University of Utah
Salt Lake City, Utah 84112

Donald E. Mains
Naval Weapons Support Center
Electrochemical Power Sources Division
Crane, Indiana 47522

S. Ruby
DOE (STOR)
Room 5E036 Forrestal Bldg., CE-14
Washington, D.C. 20595

Dr. A. J. Bard
Department of Chemistry
University of Texas
Austin, Texas 78712

Dr. Janet Osteryoung
Department of Chemistry
State University of New York
Buffalo, New York 14214

Dr. Donald W. Ernst
Naval Surface Weapons Center
Code R-33
White Oak Laboratory
Silver Spring, Maryland 20910

Mr. James R. Moden
Naval Underwater Systems Center
Code 3632
Newport, Rhode Island 02840

Dr. Bernard Spielvogel
U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709

Dr. Aaron Fletcher
Naval Weapons Center
Code 3852
China Lake, California 93555

Dr. M. M. Nicholson
Electronics Research Center
Rockwell International
3370 Miraloma Avenue
Anaheim, California

Dr. Michael J. Weaver
Department of Chemistry
Purdue University
West Lafayette, Indiana 47907

Dr. R. David Rauh
EIC Laboratories, Inc.
111 Downey Street
Norwood, Massachusetts 02062

Dr. Aaron Wold
Department of Chemistry
Brown University
Providence, Rhode Island 02192

Dr. Martin Fleischmann
Department of Chemistry
University of Southampton
Southampton SO9 5NH ENGLAND

Dr. R. A. Osteryoung
Department of Chemistry
State University of New York
Buffalo, New York 14214

Dr. John Wilkes
Air Force Office of Scientific
Research
Bolling AFB
Washington, D.C. 20332

Dr. R. Nowak
Naval Research Laboratory
Code 6171
Washington, D.C. 20375

Dr. D. F. Shriver
Department of Chemistry
Northwestern University
Evanston, Illinois 60201

END

DATE

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